ONE STEP POTASSIUM HYDROXIDE ACTIVATED AND CALCIUM OXIDE DOPED CARBON CATALYST FOR TRANSESTERIFICATION OF RICE BRAN OIL

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DEDICATION

Dedicated to my beloved parents, Muhammad Zaki bin Yacob and Zaidah binti Muhamad, to my siblings and family, to special person, Hawa Aqilah binti Hamuzan, to my friends, thank you so much for their patience, love and support.

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ABSTRACT

Palm kernel shell are a cheap and abundant biomass from palm oil industries in many tropical countries like Malaysia and Indonesia. This agricultural by-product can be a good source for the production of activated carbon. Activated carbon can be used as catalyst support in transesterification reaction to produce biodiesel. Catalyst support is important to reduce the effects of leaching in heterogeneous catalyst process. Typical preparation of activated carbon catalyst support for transesterification reaction employs a two-step process. In this study, doped activated carbon was prepared in one step using palm kernel shell activated by potassium hydroxide (KOH) and doped with calcium oxide (CaO). The modified carbon was prepared via wet impregnation method using different amount of CaO while maintaining the same percentage concentration of KOH at 25% by weight before calcined at 500^{o}C for 5 hours. The prepared carbon was then used as a heterogeneous base catalyst in transesterification reaction of rice bran oil with methanol. The modified carbon was characterized using thermogravimetric analysis (TGA), Fourier transformed infrared spectroscopy (FTIR), nitrogen adsorption analysis, field emission scanning electron microscope (FESEM), X-ray powder diffraction spectroscopy (XRD) and X-ray fluorescence spectroscopy (XRF). The basic strength of the sample was determined by back titration method. The final product of transesterification was then analysed using gas-chromatography-flame ionization detection (GC-FID) and gas-chromatography-mass spectrometry (GC-MS). XRF was employed to check the possibility of leaching of the metal catalyst into the biodiesel. TGA analysis indicates that complete calcination of palm kernel shells occur at 500^{o}C. Thus, the activation of the modified carbon was done at 500^{o}C. FTIR analysis of raw palm kernel shell shows the presence of various functional groups. However, after activation, most of the functional groups disappeared. BET surface area of 3.62 m^2/g was obtained from the 25% CaO/KOH/C due to the filling of the metal catalyst into the cavities and pores of the modified carbon. This was confirmed by washing the modified carbon several times with hot water, which later increase the BET surface area to 443.84 m^2/g. From the basicity analysis, increase in the percentage concentration of CaO increased the basicity of the prepared modified carbon. The performance of prepared modified CaO/KOH/C was identified by measuring the percentage yield of fatty acid methyl esters (FAMEs) in the transesterification of rice bran oil with methanol. The percentage yield of FAMEs for 0%, 10%, 15%, 20%, 25% and 30% CaO/KOH/C were 80.9%, 86.2%, 90.4%, 92.8%, 93.6% and 94.3%, respectively. Recyclability for the 25% CaO/KOH/C were studied and the catalyst can be reused for three consecutive runs with acceptable yield. Thus, it can be concluded that the preparation of one step KOH activated and CaO modified carbon from palm kernel shell can be used as catalyst in biodiesel production.

ABSTRAK

Tempurung kelapa sawit adalah sisa pepejal yang murah dan banyak didapati daripada industri kelapa sawit di banyak negara tropika seperti Malaysia dan Indonesia. Hasil sampingan pertanian ini boleh dijadikan sumber yang baik bagi penghasilan karbon teraktif. Karbon teraktif boleh digunakan sebagai penyokong mangkin dalam tindak balas transesterifikasi untuk menghasilkan biodiesel. Penyokong mangkin adalah penting untuk mengurangkan kesan larut resap dalam proses pemangkin heterogen. Penyediaan penyokong mangkin karbon teraktif untuk tindak balas transesterifikasi lazimnya menggunakan proses dua langkah. Dalam kajian ini, karbon aktif terdopkan telah disediakan dalam satu langkah menggunakan tempurung kelapa sawit diaktifkan dengan kalium hidroksida (KOH) dan didop dengan kalsium oksida (CaO). Karbon terubahsuai ini disediakan melalui kaedah pengisitepuan basah menggunakan jumlah CaO yang berbeza manakala peratus kepekatan KOH dikekalkan pada 25% mengikut berat sebelum dikalsin pada 500^{o}C selama 5 jam. Karbon yang disediakan kemudian digunakan sebagai mangkin bes heterogen dalam tindak balas transesterifikasi minyak bran beras dengan metanol. Karbon terubahsuai itu dicirikan menggunakan analisis termogravimetri (TGA), spektroskopi inframerah transformasi Fourier (FTIR), analisis penjerapan nitrogen, mikroskopi elektron pengimbas pemancaran medan (FESEM), spektroskopi pembelauan sinar-X (XRD) dan spektroskopi sinar-X pendarflour (XRF). Kekuatan bes sampel telah ditentukan dengan kaedah pentitratan kembali. Hasil akhir transesterifikasi itu kemudian dianalisis menggunakan kromatografi gas-pengesanan pengionan nyala (GC-FID) dan kromatografi gas-spectrometri jisim (GC-MS). XRF digunakan untuk mengesan kemungkinan logam mangkin larut resap pemangkin ke dalam biodiesel. Analisis TGA menunjukkan pengkalsinan lengkap tempurung kelapa sawit berlaku pada 500^{o}C. Dengan itu, pengaktifan karbon terubahsuai telah dijalankan pada 500^{o}C. Analisis FTIR tempurung kelapa sawit mentah menunjukkan kehadiran pelbagai kumpuan berfungsi. Walau bagaimanpun, selepas pengaktifan, kebanyakan kumpulan berfungsi itu telah hilang. Luas permukaan BET sebanyak 3.62 m^2/g diperoleh daripada 25% CaO/KOH/C disebabkan pengisian mangkin logam ke dalam rongga dan liang karbon terubahsuai. Ini telah disahkan dengan membasuh karbon terubahsuai beberapa kali dengan air panas yang kemudiannya meningkatkan luas permukaan BET kepada 443.84 m^2/g. Daripada analisis bes, peningkatan peratus kepekatan CaO telah meningkatkan kekuatan bes karbon terubahsuai yang disediakan. Prestasi karbon terubahsuai CaO/KOH/C yang disediakan telah dikenalpasti dengan mengukur peratus hasil ester metil asid lemak (FAME) dalam transesterifikasi minyak bran beras dengan metanol. Peratus hasil FAME bagi 0%, 10%, 15%, 20%, 25% dan 30% CaO/KOH/C ialah masing-masing 80.9%, 86.2%, 90.4%, 92.8%, 93.6% dan 94.3%. Penggunaan semula bagi 25% CaO/KOH/C telah dikaji dan mangkin itu boleh digunakan semula bagi tiga tindak balas berturut-turut dengan hasil yang boleh diterima. Dengan itu, boleh disimpulkan bahawa penyediaan karbon satu langkah teraktifkan-KOH dan terubahsuai-CaO daripada tempurung kelapa sawit boleh digunakan sebagai mangkin dalam pengeluaran biodiesel.

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ABBREVIATIONS

AC	-	Activated Carbon
BET	-	Brunauer-Emmett Teller
CaO	-	Calcium Oxide
CaO/KOH/C	-	Potassium hydroxide and calcium oxide modified carbon
CO_2-TPD	-	Carbon dioxide-Temperature Program Desorption
FAME	-	Fatty acid methyl ester
FESEM	-	Field Emission Scanning Electron Microscope
FID	-	Flame Ionization Detector
FTIR	-	Fourier Transform Infrared
GC-FID	-	Gas Chromatography- Flame Ionization Detector
GC-MS	-	Gas Chromatography Mass Spectrometer
KOH	-	Potassium hydroxide
PKS	-	Palm kernel shell
RBO	-	Rice bran oil
TGA	-	Thermogravimetric Analyzer
XRD	-	X-ray Powder Diffraction
XRF	-	X-ray Fluorescence

APPENDICES (A-E)

CHAPTER 1

INTRODUCTION

1.1 Background of Research

Activated carbon (AC) is a black solid substance that can be present in granular or powder form. It is a carbonaceous material with a well-developed porosity and high mechanical strength. AC is known as one of the most versatile materials and can be used as a support and a good adsorbent. Moreover, the physical and chemical properties of activated carbon can be easily tailored to meet various specific needs. Its high surface area and well developed porosity make it desirable for several important industrial applications. Many industries including waste water treatment (Ribeiro *et al.*, 2015), chemical recovery process (Kaushik *et al.*, 2017) and separation treatment (Cermakova *et al.*, 2017) are using activated carbon in their process. Activated carbon can also be used as catalyst support (Samad *et al.*, 2017). Moreover, activated carbon can be used in high temperature and high pressure reactions because its adsorptive properties were not affected greatly at high temperature and pressure (Nowicki and Pietrzak, 2017).

As the production and regeneration of commercial AC are still expensive, there are many continuing researches for potential low cost raw material and methods (Acikyildiz *et al.*, 2014; Lounasvuori *et al.*, 2018; Wei *et al.*, 2018) . There is no limitation on the raw material that can be used to produce activated carbon. Almost any carbonaceous material with low organic volatile and high carbon content are desirable and can be converted into AC. The raw material and method used in the production of activated carbon would determine the quality of the activated carbon produced. Usually, the material used for the preparation of AC are fossil fuel generated hydrocarbon and lignocelluloses from biomass wood or some agricultural waste. However, lignocellulose agricultural wastes are regarded as the most suitable raw materials for the production of AC. The advantage of using agricultural by product as raw material for manufacturing AC is that these raw materials are potentially less expensive to manufacture and environmentally conscious (El-Sayed *et al.*, 2014). Furthermore, AC produced from this agricultural waste can also facilitate disposal problem of these waste products. Various studies had been proposed regarding the production of activated carbon from agricultural by-products including coconut shell (Sun *et al.*, 2017), pistachio nut shell (Niksiar and Nasernejad, 2017), hazelnut shell (Kwiatkowski and Broniek, 2017) sugar cane bagasse (Kaushik *et al.*, 2017), olive stones (Bohli *et al.*, 2015), macadamia nut shell (Rodrigues *et al.*, 2013) and palm kernel shell (Kundu *et al.*, 2015).

One of the most valuable crops in Malaysia is palm oil. Malaysia is accounted as one of the biggest producers and exporters of palm oil products. Recently, Malaysia had generated a total production of 21.5 million tonnes of palm oil export commodities and it is projected to reach 25.6 million tonnes of crude palm oil per year by 2050 (Umar *et al.*, 2018). In Malaysia, palm oil industries are the main producers of the abundant amount of lignocellulosic biomass. At present, large quantities of palm oil biomass exceeding 50 million tonnes, is produced anually (Liew *et al.*, 2015). The by-product of palm milling process include palm pressed fibre, palm kernel shell and empty fruit bunch (Uemura *et al.*, 2011). Palm kernel shell shows great potential as a precursor for activated carbon production compared to other biomass such as wood, nutshell and coconut shell. The natural characteristics of palm kernel shell make it suitable for AC production since it contains high oxygen and carbon content. These characteristics are known to produce good AC. Although currently some quantity of PKS are normally used as boiler feed to generate steam in the palm oil processing plant, the residue still remain as a surplus which is discarded by open burning or left in mill premises itself (Foo and Hameed, 2012). Therefore, it is profitable if the PKS are also be exploited to produce higher value product such as activated carbons.

With the aim of overcoming the accumulation of agricultural wastes, several attempts have been made to convert these biomasses into value added products such as pulp and paper, particle boards, pelleted fuels, soil conditioners, animal feedstock, organic acids, activated carbon, plywood and fertilizer (Wahid, 2011; Oviasogie *et al.*, 2013). In addition, converting these agricultural waste into activated carbon is significant due to the increasing market demand on this carbonaceous material that is estimated to reach about 2.1 metric tonnes by year 2018 (Maneerung *et al.*, 2016a). In another point of view, conversion of these agricultural waste can elevate financial income and promote employment opportunities.

Generally, the preparation of AC may either use a physical or chemical activation method. Physical activation is a two-step process which are the carbonization step and activation step. In physical activation process the raw material is first carbonized followed by activation by steam or carbon dioxide. Whereas chemical activation can be done in a single step by thermal decomposition of raw material impregnated with chemical activating reagents such as zinc chloride ($ZnCl_2$), phosphoric acid (H_3PO_4), potassium hydroxide (KOH) and sodium hydroxide (NaOH). Chemical activation offers several advantages and disadvantages compared to physical activation. The main advantages are that it can produce a much higher yield at lower activation temperature with less activation time and it can be done in a single step. Moreover, it has a better pore development compared to physical activation process. However, among the disadvantages of chemical activation process is that the chemical activating agents are much more expensive and it is also requires a washing step after activation to remove the chemical activator (Maciá-Agulló *et al.*, 2004).

Currently, the world is facing a vital calamity with regards to energy and fossil fuel. This is because the demand for energy for economic development and population growth is increasing in the last few years. The most important concern is that energy source is typically derived from fossil fuel. This fossil fuel is non-renewable, it has a limited supply and one day it will be depleted. Thus, new source of energy is needed in order to reduce the energy dependence on fossil fuel and also produce less net pollution to the environment. Biodiesel being renewable and environment friendly are considered a feasible route to resolve this problem and has been commercialized as a substitute for petroleum base diesel. In addition, biodiesel is completely miscible with petroleum diesel and can be used as blending stock of petroleum base fuel.

The study of biodiesel as an alternative, nontoxic, biodegradable and renewable fuel is rapidly growing due to the dwindling of fossil fuel resource and the increasing demand for energy. Biodiesel is a non petroleum-based fuel having similar fuel properties as petroleum diesel usually produced from renewable resource such as vegetable oil or animal fat.

Biodiesel consists of mixture of long chain fatty acid alkyl esters mainly fatty acid methyl ester (FAME) or fatty acid ethyl ester (FAEE) (Moser, 2009). The use of biodiesel in conventional diesel engines can reduce the emission of unburnt hydrocarbon, carbon monoxide and particulate because vegetable oil esters contain 10-11% oxygen by weight that can enhance combustion compared to hydrocarbon base diesel (Agarwal, 2007). In general, in terms of power, wear, efficiency and emissions, biodiesel fuels are a viable alternative to petroleum based diesel. There are a few methods for the production of biodiesel including direct use and blending, micro emulsion, thermal cracking (pyrolysis) and transesterification.

The most successful ways of producing biodiesel is by transesterification reaction because it is cost effective and simpler method (Ma and Hanna, 1999). Biodiesel production through transesterification, also known as alcoholysis, involves a reaction between triglyceride with alcohol to produce alkyl ester and glycerol. The alcohol used is usually methanol or ethanol and the reaction usually needs a catalyst to enhance the reaction. The reaction consists of a series of consecutive reversible reactions to produce 3 mol of fatty acid alkyl ester and glycerol as a by-product. The ovearall reaction equation is shown in Figure 1.1. Generally, excess alcohol is needed to shift the forward reaction (Badday *et al.*, 2014). Triglyceride and the alcohol are not miscible, thus the presence of catalyst is necessary in transesterification reaction in order to achieve high conversion without any harsh reaction condition. Therefore, selection of a right catalyst is very important to get the highest conversion of vegetable oil into biodiesel (Dias *et al.*, 2012).

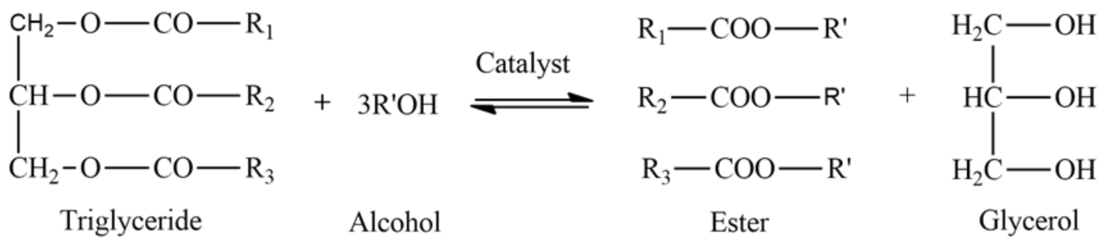

Figure 1.1: Overall transesterification reaction of triglyceride with alcohol (Ejikeme *et al.*, 2010)

There exist a wide variety of catalyst for transesterification reactions that can be either acid, base or even mixed oxide that can be present in homogenous or heterogeneous state. Currently, biodiesel is produced industrially by the transesterification of vegetable oil with methanol using a homogeneous base catalyst based on NaOH and KOH to achieve high conversion close to 100% at reaction temperature of around 60^{o}C under less than 3 hours (Alcañiz-Monge *et al.*, 2013). Although the homogeneous catalysis process can produce high yield of biodiesel under mild reaction conditions with fast reaction rate, this process however involves different stages of purification of the biodiesel and the glycerol and it generates large amount of waste water effluents. To minimize the problems associated with this approach, heterogeneous catalytic process has been proposed. The main objective of using heterogeneous catalyst are to simplify the production process as well as to reduce the purification cost and environmental impacts. Heterogeneous catalyst offer several advantages over homogenous catalyst such as elimination of washing step to isolate the product, less corrosive, safer, and environmental friendly, which would reduce the total capital and energy cost (Agarwal *et al.*, 2012). Furthermore, heterogeneous catalyst can be reused and would not have to be continuously added.

A variety of solid bases has been reported to be a good heterogeneous catalyst for biodiesel transesterification. Metal oxides, hydroxides and alkoxides has been developed to catalyse the transesterification reaction of vegetable oil for biodiesel production. In the transesterification of vegetable oil or animal fat into biodiesel process, activated carbon can be used as a heterogeneous support catalyst. The main concern for heterogeneous catalysis is to achieve high surface area to enhance the reactivity and selectivity. This can be achieved by introducing a support to the catalyst to disperse the catalyst species to obtain optimal performance. Most support can be tailored to meet its specific need either by functionalization or doping process. Support for heterogeneous catalysis can be alumina, zeolite, carbon nanofibre, activated carbon and metal oxide such as TiO_2, and MnO_2 (Bagheri *et al.*, 2014). Occasionally, the preparation of heterogeneous catalyst can contribute to an additional cost. The use of simple, safe and low cost in the preparation of the catalyst can be attractive.

Therefore, in this study, an active and reusable catalyst based on modified carbon from palm kernel shell was produced through one step activation process using potassium hydroxide (KOH) as the activating agent and calcium oxide (CaO) as the catalyst to further increase the basicity of the modified carbon. The prepared modified carbon is then tested as a potential heterogeneous base catalyst in the synthesis of biodiesel from rice bran oil with methanol under a mild reaction conditions. In addition, this study also aimed to investigate the possibility of increasing the strength of basic sites of KOH and CaO mixed in order to improve their performance in the transesterification reaction. These two base catalyst was chosen due to their high basicity which is important for catalysing heterogeneously catalysed transesterification reaction.

1.2 Problem Statement

At present, the main industrial biodiesel production method is the alkali catalysed homogenous process between vegetable oil and methanol. According to the National Biofuel Policy report from 2016, the implementation of the 10% biodiesel blend (B10) planned for July 1st 2016 has been postponed to early 2017. This is due to the claim that a higher blend biodiesel above 7% could damage the engines (Zabid *et al.*, 2018). This is due to the contamination of the biodiesel with the catalyst because of poor separation between the biodiesel and the catalyst. Furthermore, a large amount of water is needed in order to remove the homogenous catalyst that is dissolved into the biodiesel and this washing step sometimes may lead to saponification reaction. To overcome the problem connected with the use of homogenous catalyst, attentions was directed toward the development of heterogeneous catalyst for biodiesel production. Heterogeneous catalyst are easy to separate from the reaction mixture, can be reused and will prevent catalyst contamination of the biodiesel (Kaur and Ali, 2014).

Among the different heterogeneous base catalyst, CaO is one of the most active. It is widely used in the transesterification reaction of vegetable oil with methanol to produce biodiesel. However, CaO present limitation in terms of leaching, because some of the Ca^{2+} is leached to the alcoholic phase, which would cause an additional process of neutralization and cleaning of the leached species. The leached species can reduce the catalytic property and reusability of the catalyst. Furthermore, the accumulation of these leached species also can harm the mechanical properties of the diesel engine. As a result, mixed oxide or a suitable support are used to increase the interaction between active phase and prevent leaching making the catalyst more stable (Syamsuddin *et al.*, 2015). In this study, CaO catalyst is supported on the modified carbon to reduce the leaching effect.

Generally, preparation of catalyst supported activated carbon involve two-step processes in which the activated carbon was first prepared followed by the impregnated of the catalyst species. Furthermore, in chemical activation process, the activating agent is left as impurities after heat treatment and sometimes may affect the chemical properties of the resulting activated carbon. Therefore, washing steps is required to remove the remaining chemical activator, which is time consuming and sometimes lead to an environmental issue.

Rice is one of the major staple food in Malaysia making paddy farm agriculture another major industry in Malaysia. Rice bran oil is produced from rice bran which is mostly considered as agricultural waste in paddy farms plantations. As rice is being produced largely in Malaysia, the utilisation of wasted rice bran to obtain oil for biodiesel production will help to increase the value of rice and help to improve local economies. In addition, the use of rice bran oil from agricultural waste can help to reduce the food versus fuel competition which may result in the cost or food shortage (Zhao *et al.*, 2013).

Hence, in this study, palm kernel shell modified carbon was produced by means of chemical activation with KOH and CaO is doped into the activated carbon in a one-step activation process. In addition, instead of washing the activating agent, the impurities left in the activated carbon will be used as a base catalyst.

1.3 Objective of Research

The objective of this study are :

1. To prepare and characterize potassium hydroxide and calcium oxide modified carbon catalyst (CaO/KOH/C) via one step activation process.
2. To apply the prepared catalyst as base heterogeneous catalyst in the transesterification reaction of rice bran oil with methanol.
3. To study the effect of leaching and recyclability of the catalyst.
4. To characterize the composition of biodiesel produce

1.4 Scope of the Research

The scope of the present study can be divided into 3 major aspects. The first aspect is the preparation of the potassium hydroxide and calcium oxide modified carbon catalyst by one step activation method. The activated carbon catalyst is prepared by impregnating different concentration of CaO at 0%, 10%, 15%, 20% and 25% concentration by weight while maintaining the concentration of potassium hydroxide at 25% concentration by weight into the palm kernel shell. Then, it is calcined at 500^{o}C for 5 hours.

The second aspect is to characterize the prepared catalyst. Thermogravimetry analysis (TGA) for raw palm kernel shell was applied in order to determine the activation temperature. The prepared CaO/KOH/C catalyst were then characterized via Fourier Transform Infrared (FTIR), X-ray Powder Diffraction (XRD), X-ray Fluorescence (XRF) and Nitrogen Adsorption Analysis. Field Emission Scanning Electron Microscope (FESEM) analysed their surface morphology while energy dispersive x-ray (EDX) was used to identify the elemental composition of the prepared catalyst. The basicity and basic strength of the catalyst were analysed by back titration method and Temperature Programmed Desorption (CO_2-TPD). The possibility of catalyst leaching into biodiesel will be analysed by determining the presence of potassium and calcium in the biodiesel product via X-ray Fluorescence (XRF).

The third scope of this study is to test the prepared CaO/KOH/C catalyst in the transesterification of rice bran oil with methanol. The analysis and yield of biodiesel were performed Gas Chromatography-Flame Ionization Detector (GC-FID) and the confirmation of the methyl esters were identified by Gas Chromatography Mass Spectrometer (GC-MS). The recyclability of the catalyst was done by washing the catalyst with hexane after the transesterification reaction and drying in oven overnight. The catalyst was then used in the next cycle of transesterification reaction and the effect of leaching was also studied.

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